Quantitative Differentiation between Natural Capsaicin (8-Methyl-*N*-vanillyl-6-nonenamide) and Vanillyl-*n*-nonanamide

SIR: The Joint Committee on Methods of Assay of Crude Drugs of the Pharmaceutical Society and the Society of Analytical Chemistry of Great Britain [*Analyst* **84**, No. 1003, 603 (1959)] have recently reviewed the existing methods of capsaicin analysis. They reported that there is no satisfactory chemical method available to distinguish between natural capsaicin and the synthetic compound, vanillyl-*n*-nonanamide. The ultraviolet absorption characteristics of both compounds are practically identical. The reaction products of both the synthetic compound and natural capsaicin with diazobenzenesulfonic acid have an absorption maximum at 480 mμ. They concluded that the diazo method is not adequate and that some acceptable procedure should be developed.

Reported below is an infrared spectrophotometric procedure by which natural capsaicin and vanillyl-*n*-nonanamide can be distinguished and the proportion of each component in a mixture can be evaluated quantitatively. The method is based on the infrared absorption of natural capsaicin at 970 cm.$^{-1}$ which is due to a trans double bond present in the fatty acid moiety of the natural compound.

Infrared spectra from 900 to 1100 cm.$^{-1}$ of a chloroform solution containing 150 mg. per ml. of natural capsaicin and a solution containing 150 mg. per ml. of vanillyl-*n*-nonanamide measured against pure chloroform are shown in Figure 1. The data were obtained with a Perkin-Elmer Model 21 instrument equipped with 0.2-mm. cells and using a constant mechanical slit width of 151 microns. Several mixtures of the two components were subsequently prepared and the content of natural capsaicin evaluated by the base line method. Each absorbance value was obtained in duplicate. The per cent of natural capsaicin calculated from the absorbance data is shown in Table I.

The composition of mixtures of natural capsaicin and vanillyl-*n*-nonanamide can be determined more conveniently but with a slight loss of accuracy by measuring the ratio of the base line absorbances of the 970-cm.$^{-1}$ band and the 1040-cm.$^{-1}$ band. The latter band is of equal intensity in both compounds. If the ratio method is used, the chloroform solutions of capsaicin should be of lower concentration (ca. 100 mg. per ml.) to permit the measurement of the intensity of the very strong 1040-cm.$^{-1}$ band. The ratio A_{970}/A_{1040} (where A_{970} and A_{1040} are base line absorbances) was 0.43 for the crystalline capsaicin prepared in this laboratory. The per cent natural capsaicin in an unknown mixture is calculated by:

$$\% \text{ natural capsaicin} = \frac{A_{970}}{A_{1040}} \times \frac{1}{0.43} \times 100$$

Thus, the composition of mixtures of natural capsaicin and vanillyl-*n*-nonanamide can be determined quantitatively by infrared absorption spectroscopy. It should also be possible to distinguish between capsaicin and similar synthetic pungent compounds which do not contain a trans double bond. For example, vanillyl-*n*-10-undecenamide, which has a terminal double bond, has the same absorptivity at 1040 cm.$^{-1}$ but no absorption band at 970 cm.$^{-1}$. Column chromatography or ether-alkali extraction methods, which are used for the extraction and purification of capsaicin, in conjunction with infrared absorption can be used to isolate and identify pungent components extracted from red pepper.

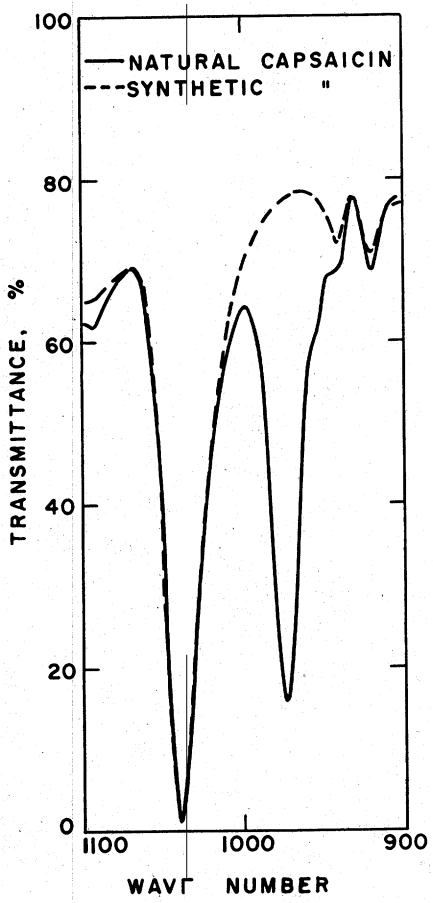

Figure 1. Infrared spectra

Table I. Per Cent of Natural Capsaicin Added and Calculated from the Infrared Absorbance Data

% Natural Capsaicin	
Added	Calculated
10	10.2
20	18.9
40	39.6
60	61.0
80	81.4

P. R. DATTA
H. SUSI

Eastern Utilization Research and Development Division
Agricultural Research Service
United States Department of Agriculture
Philadelphia 18, Pa.

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